# Growth and Doping of SiC-Thin Films on Low-Stress, Amorphous $Si_3N_4$/Si Substrates for Robust Microelectromechanical Systems Applications

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The $N_2$-doped 3C-SiC thin films have been grown by low-pressure, chemical vapor deposition (LPCVD) on amorphous $Si_3N_4$/p-Si (111) substrates using the single, organosilane-precursor trimethylsilane [$(CH_3)_3SiH$]. The effects of $N_2$ flow rate and growth temperature on the electrical properties of SiC films were investigated by Hall-effect measurements. The electron-carrier concentration is between $10^{17}$–$10^{18}$/cm$^3$. The lowest resistivities at 400 K and 300 K are $1.12 \times 10^{-2}$ and $1.18 \times 10^{-1}$ cm, respectively. The corresponding sheet resistances are 75.02 Ω/□ and 790.36 Ω/□. The SiC film structure was studied by x-ray diffraction. The 3C-SiC films oriented in the ⟨111⟩ direction with a 2θ peak at 35.5° and line widths between 0.18–0.25° were obtained. The SiC/$Si_3N_4$ interface is very smooth and free of voids. The fabrication of microelectromechanical (MEMS) structures incorporating the SiC films is discussed.

**Key words:** CVD, LPCVD, SiC, 3MS, MEMS

## INTRODUCTION

Silicon carbide is recognized[1] as a wide-bandgap semiconductor with superior thermal, electrical, mechanical, and chemical properties for the production of high-temperature, high-power, and high-frequency electronic devices. In comparison to Si, SiC, with its high Young's modulus and toughness, chemical inertness, and radiation resistance, makes an excellent candidate for the fabrication of microelectromechanical systems (MEMS) devices that need to function at high temperature as well as in harsh environments. In comparison to diamond, attractive features of SiC are that it can be doped both p- and n-type, and it allows a natural oxide to be grown on its surface for the purpose of certain device fabrication.[2]

The MEMS applications require that large-area, uniform SiC films are formed on insulating or sacrificial layers,[3,4] such as $SiO_2$, $Si_3N_4$, and polycrystalline Si (poly-Si). Because of the decomposition of $SiO_2$ at an SiC growth temperature above 1100°C, oxygen contamination during SiC growth may occur and can affect the electrical properties of SiC films for the device purpose. The $Si_3N_4$ then becomes more suitable as an insulating layer for SiC-MEMS devices. Chemical vapor deposition (CVD) is an excellent tool for conformal growth on MEMS structures with complex three-dimensional topology. The SiC-device definition and processing are well established. We have previously reported[5] that the single, organosilane-precursor trimethylsilane [$(CH_3)_3SiH$ or 3MS] produces excellent 3C-SiC films while being nonpyrophoric, noncorrosive, and easier to handle than the conventional $SiH_4/C_3H_8/H_2$ gas system used for SiC growth. Our previous results with 3MS show that SiC films grown on Si substrates[6] can be in situ doped with $N_2$ and that undoped films can be grown successfully on $Si_3N_4$ layers[3] on Si substrates.

In this paper, the electrical properties of in situ $N_2$-doped 3C-SiC films grown on low-stress, amorphous $Si_3N_4$/Si (100) substrates using 3MS are investigated by Hall-effect measurements, while structural characterization of these films has also been investigated by x-ray diffraction (XRD), scanning electron microscopy (SEM), and atomic force microscopy (AFM). The resistivity and sheet resistance are measured as a function of $N_2$ flow rate. In addition, the initial fabrication of MEMS structures by inductively coupled plasma (ICP), dry etching has shown that low-pressure (LP) CVD conformal growth of SiC on low-stress, amorphous $Si_3N_4$ layer is successful.

(Received September 8, 2001; accepted December 28, 2001)

# Report Documentation Page

*Form Approved*
*OMB No. 0704-0188*

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| 1. REPORT DATE | 2. REPORT TYPE | 3. DATES COVERED |
|---|---|---|
| **2002** | | **00-00-2002 to 00-00-2002** |

| 4. TITLE AND SUBTITLE | 5a. CONTRACT NUMBER |
|---|---|
| **Growth and Doping of SiC-Thin Films on Low-Stress, Amorphous Si3N4/Si Substrates for Robust Microelectromechanical Systems Applications** | 5b. GRANT NUMBER |
| | 5c. PROGRAM ELEMENT NUMBER |
| 6. AUTHOR(S) | 5d. PROJECT NUMBER |
| | 5e. TASK NUMBER |
| | 5f. WORK UNIT NUMBER |
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)<br>**University of Cincinnati,Nanoelectronics Laboratory,Cincinnati,OH,45221-0030** | 8. PERFORMING ORGANIZATION REPORT NUMBER |
| 9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) | 10. SPONSOR/MONITOR'S ACRONYM(S) |
| | 11. SPONSOR/MONITOR'S REPORT NUMBER(S) |

12. DISTRIBUTION/AVAILABILITY STATEMENT
**Approved for public release; distribution unlimited**

13. SUPPLEMENTARY NOTES

14. ABSTRACT

15. SUBJECT TERMS

| 16. SECURITY CLASSIFICATION OF: | | | 17. LIMITATION OF ABSTRACT | 18. NUMBER OF PAGES | 19a. NAME OF RESPONSIBLE PERSON |
|---|---|---|---|---|---|
| a. REPORT | b. ABSTRACT | c. THIS PAGE | | | |
| **unclassified** | **unclassified** | **unclassified** | | **5** | |

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## EXPERIMENTAL

The SiC films were grown in a LPCVD system. The details of the system can be found in Ref. 6. The base pressure in the chamber is $3 \times 10^{-3}$ torr, and the operating pressure is 13–14 torr. The gas purity is 99.999% for hydrogen, 99.995% for 3MS, and 99.999% for nitrogen. For all SiC-growth experiments reported, the hydrogen flow rate is 1 slm, and the 3MS flow rates range from 20–40 sccm, while nitrogen flow rates vary from 0–10 sccm. The growth temperature is in the range of 1100–1250°C, the growth time is 3–6 min, and temperature ramp rate is 25°C/sec. The temperature is monitored and controlled by an optical pyrometer. The crystal structure of the SiC films was characterized by XRD spectra. The surface morphology of the SiC films and the SiC/$Si_3N_4$ interface was examined by SEM. The surface roughness of the SiC films was studied by AFM. The SiC-film thickness of samples used for Hall measurements ranged from 1–2 μm. Electrical properties of grown SiC films were investigated using MMR Corporation's (California, USA) Hall-effect measurement system with a magnetic field up to 1.2 T. Electrical contacts were made by depositing Ni on the four corners of the sample using electron-beam evaporation followed by annealing at 900°C for 5 min. The quality of the electrical contacts was evaluated by checking the linearity of their current-voltage (I-V) relation. The magnetic field was set at 1 T (10,000 Gauss) for all Hall-effect measurements.

## RESULTS AND DISCUSSION

### Structure

The θ-2θ XRD spectra for SiC films grown on $Si_3N_4$/Si at different growth temperature are shown in Fig. 1. The spectra were normalized by the thickness of the SiC films. These spectra show that polycrystalline SiC films oriented in the ⟨111⟩ direction were obtained at growth temperatures from 1150–1250°C. No other orientations of SiC films were detected. The normalized XRD intensity of the SiC (111) peak is plotted in Fig. 2 as a function of growth temperature. As expected, the SiC ⟨111⟩ signal increased with temperature from 1100°C to 1200°C but decreased when the temperature was increased to 1250°C. To understand the cause for this decrease, we investigated the properties of the $Si_3N_4$ layer under annealing conditions chosen to simulate the SiC growth process. Figure 3 contains XRD spectra of the $Si_3N_4$ layers annealed at temperatures from 1100–1250°C. The spectra indicate that the $Si_3N_4$

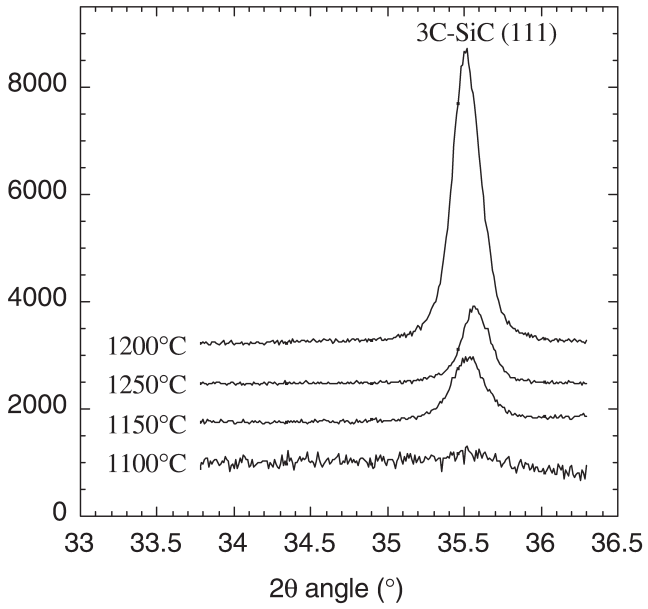


Fig. 1. Normalized XRD spectra for SiC films grown on Si3N4/Si (100) at different temperatures. Flow rates: 30 sccm 3MS and 1 sccm N2.

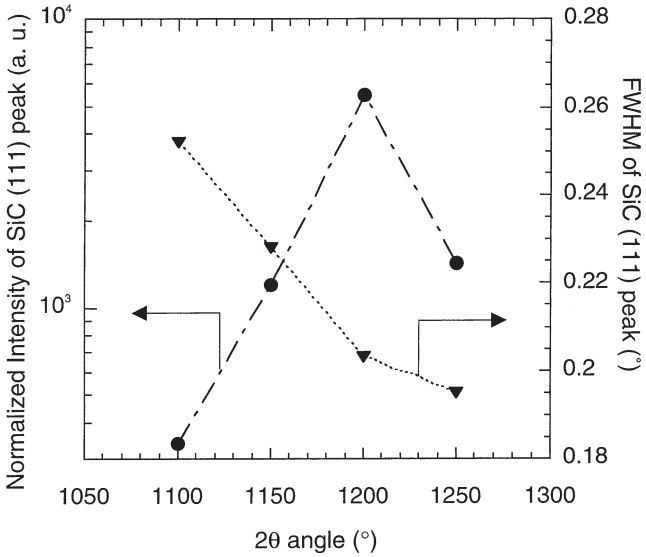


Fig. 2. Normalized XRD intensity of the SiC (111) peak as a function of growth temperature. Flow rates constant at 30 sccm 3MS and 1 sccm $N_2$.

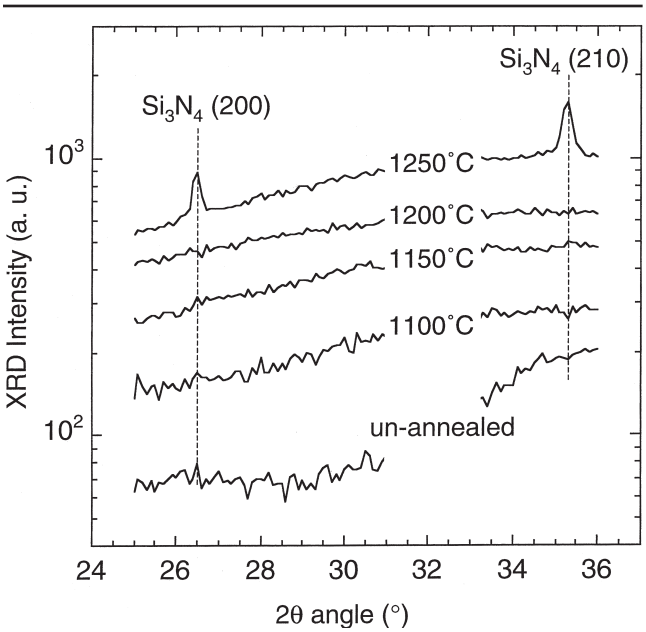


Fig. 3. The XRD spectra of the $Si_3N_4$ layer unannealed and annealed at the temperatures used for SiC growth.

layer remains amorphous after anneals up to 1200°C. However, after the 1250°C anneal, two XRD peaks with 2θ angles at 26.437° and 35.350° are observed. The peaks correspond to α-$Si_3N_4$ ⟨200⟩ and ⟨210⟩ directions. We suspect that the crystallization of the $Si_3N_4$ is the reason for the decrease of the SiC ⟨111⟩ XRD signal at a growth temperature of 1250°C. Interestingly, the full width at half maximum (FWHM) of the SiC (111) XRD peak decreases monotonically with increasing SiC-growth temperature, ranging from 0.25° at 1100°C to 0.18° at 1250°C.

The effect of the 3MS flow rate on the crystallinity of the SiC films was also investigated. Figure 4 indicates that increasing the 3MS flow rate in a certain range at a growth temperature of 1200°C results in a significant increase in the XRD intensity and a slight decrease in the FWHM of the SiC (111) peak. In the following Hall-effect measurement, we investigated how the crystallinity affects the resistivity and sheet resistance of SiC films grown under various conditions.

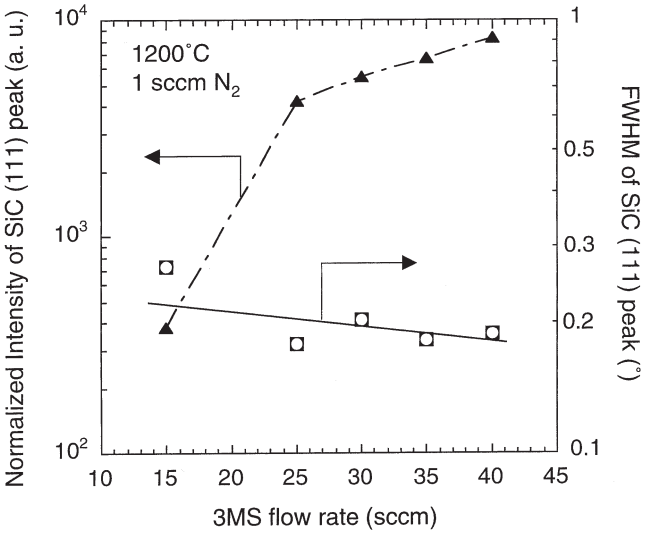


Fig. 4. Normalized XRD intensity and FWHM of the SiC (111) peak as a function of 3MS flow rate. Growth temperature: 1200°C, and $N_2$ flow rate: 1 sccm.

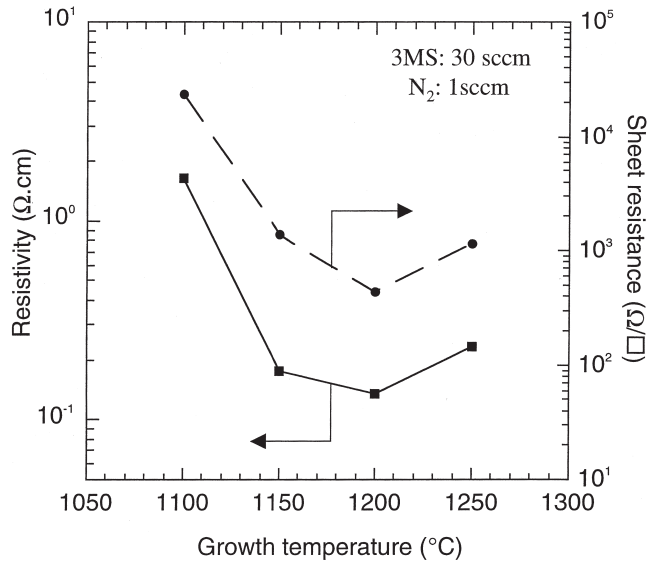


Fig. 5. Resistivity and sheet resistance of SiC films at 300 K as a function of growth temperature. Flow rates: 30 sccm 3MS and 1 sccm $N_2$.

## Electrical Properties

Figure 5 shows the resistivity and sheet resistance of SiC films as a function of growth temperature with flow rates of 30 sccm 3MS and 1 sccm

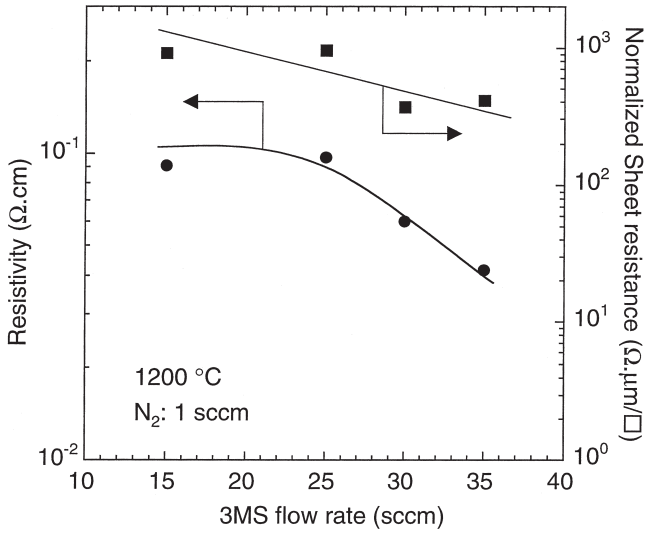


Fig. 6. Resistivity and sheet resistance of SiC films at 300 K as a function of 3MS flow rate. Growth temperature: 1200°C, and $N_2$ flow rate: 1 sccm.

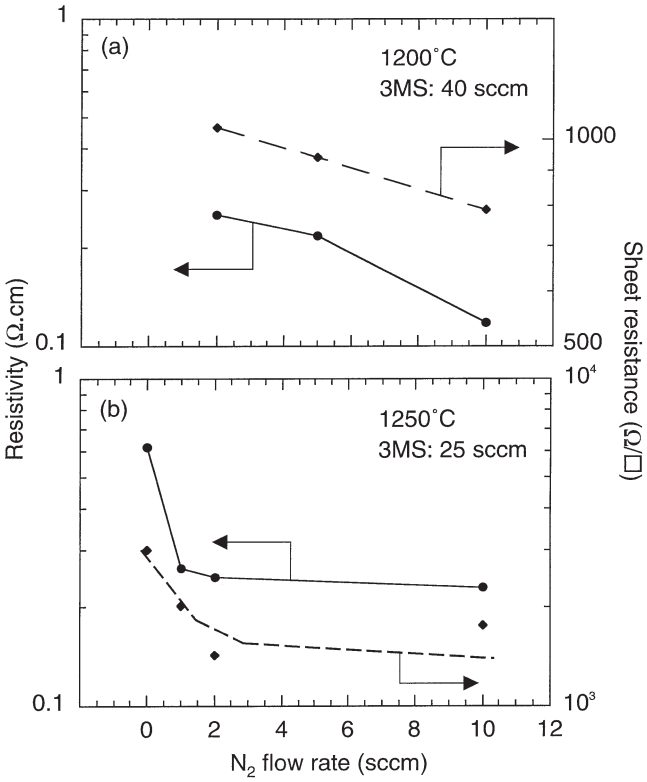


Fig. 7. Resistivity and sheet resistance of SiC films at 300 K as a function of $N_2$ flow rate: (a) growth temperature of 1200°C 3MS and flow rate of 40 sccm and (b) growth temperature of 1250°C 3MS and flow rate of 25 sccm.

$N_2$. The lowest resistivity and sheet resistance were obtained at 1200°C, which also resulted in the best crystallinity as seen in the XRD data of Figs. 1 and 2. By increasing the 3MS flow rate, we observed that the resistivity and sheet resistance decreased, as shown in Fig. 6, while the normalized XRD intensity of the SiC (111) peak increased (Fig. 4). These results are consistent with the general observation that improvements in the semiconductor yield improved electrical properties.

The effect of the $N_2$ flow rate on the SiC electrical properties was studied for two sets of growth conditions. The first used a lower 3MS flow rate (25 sccm) at 1250°C, while the second used a higher 3MS flow rate (40 sccm) at 1200°C. The $N_2$ flow rate was varied from 0–10 sccm. For both sets of operating parameters, the data of Fig. 7 shows the same trend of decreasing resistivity and sheet resistance with increasing $N_2$ flow rate. This indicates that an increasing number of N atoms incorporated into the SiC film helps to lower the resistivity and sheet resistance.

## Morphology

The surface and interface quality was examined by SEM. As shown in Fig. 7, the surface morphology of SiC film becomes worse with increasing growth temperature, while the SiC/$Si_3N_4$ interface is very smooth and free of voids for all samples. Clearly, the $Si_3N_4$ layer prevents Si out-diffusion from the substrate and subsequent reaction with C or H from the gas phase, which causes the formation of voids at the SiC/Si interface during growth directly on a Si substrate or layer. Figure 9 compares SiC-growth rate and surface roughness as a function of growth temperature. The root-mean square roughness measured by AFM and the growth rate both increase with increasing growth temperature, which conforms to the results of surface morphology observed by SEM. The SiC film thickness, measured using cross-sectional SEM, ranges from 0.7–3 μm. The growth rate obtained at the optimum temperature of 1200°C is about 0.5 μm/min, while the RMS surface roughness is approximately 40 nm.

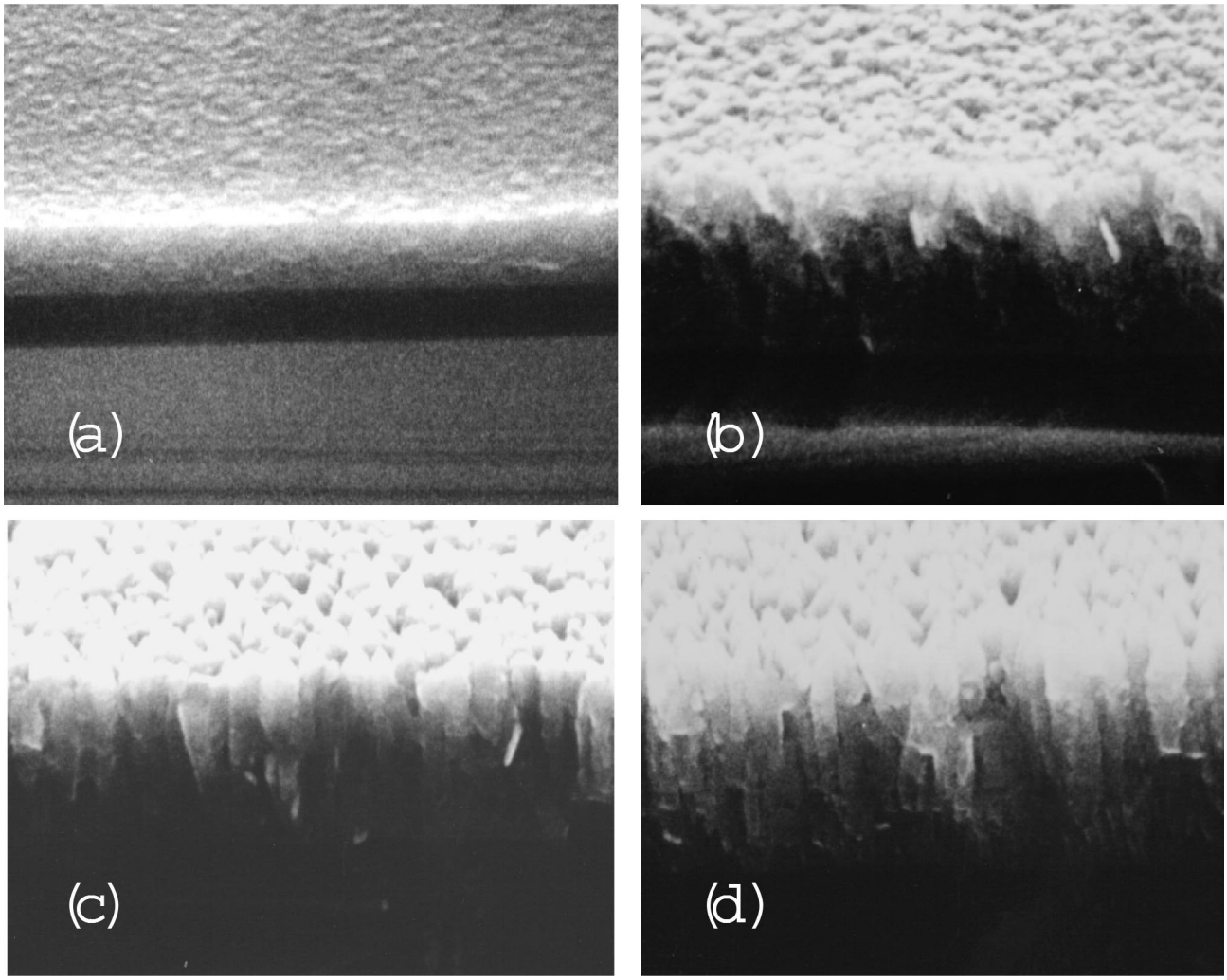


Fig. 8. The SEM micrographs of SiC-thin films grown on $Si_3N_4$/Si structures with 30 sccm 3MS and 1 sccm $N_2$ at the following temperatures: (a) 1100°C, (b) 1150°C, (c) 1200°C, and (d) 1250°C.

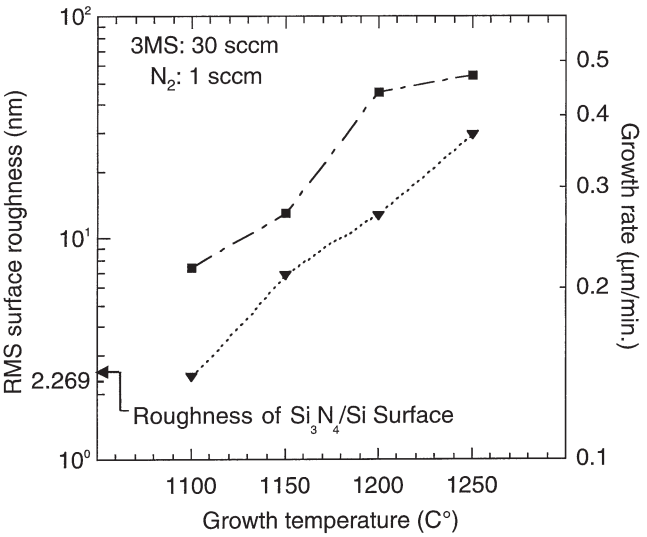


Fig. 9. Growth rate and surface roughness of SiC films versus growth temperature. Flow rates: 30 sccm 3MS and 1 sccm $N_2$.

## Microelectromechancial Fabrication

The SiC-film samples grown on $Si_3N_4$/Si (100) substrate materials were patterned and processed in initial MEMS fabrication efforts. The ICP etching using $NF_3$/Ar mixtures has shown that LPCVD conformal growth of SiC on low-stress, amorphous $Si_3N_4$ layer is successful. Vertical sidewalls, indicating high-anisotropy $NF_3$/Ar etching, have been obtained under specific etching conditions. Simple MEMS structures, such as cantilevers, lateral resonators, and resonating membranes, have been fabricated. The film stress was measured by using bent-beam strain gauges. The deflecting angle of our bent-beam is 6°. The residual stress generated by a bimorph involving a SiC-thin film has been estimated at ~188 MPa. Lateral deflection of the supporting beam-vernier structures correlates directly to the stress in the deposited film. A Young's modulus for our SiC films of E = 426 GPa, used for stress calculations, was obtained from MEMS resonance measurements for the SiC films.

## SUMMARY

We have reported on the fabrication and properties of SiC structures for high-temperature, MEMS applications. The MEMS device requires the growth and doping of SiC-thin films on amorphous $Si_3N_4$ layers on Si substrates. The SiC films were grown by CVD with the single organosilane trimethylsilane, while the in situ doping was performed by addition of N2 into the reaction chamber. A resistivity of 75 Ω/□ was measured for the doped SiC film at 400 K when operating the structure at 400 K. The MEMS structures, such as cantilevers, lateral resonators, and membranes, were successfully fabricated.

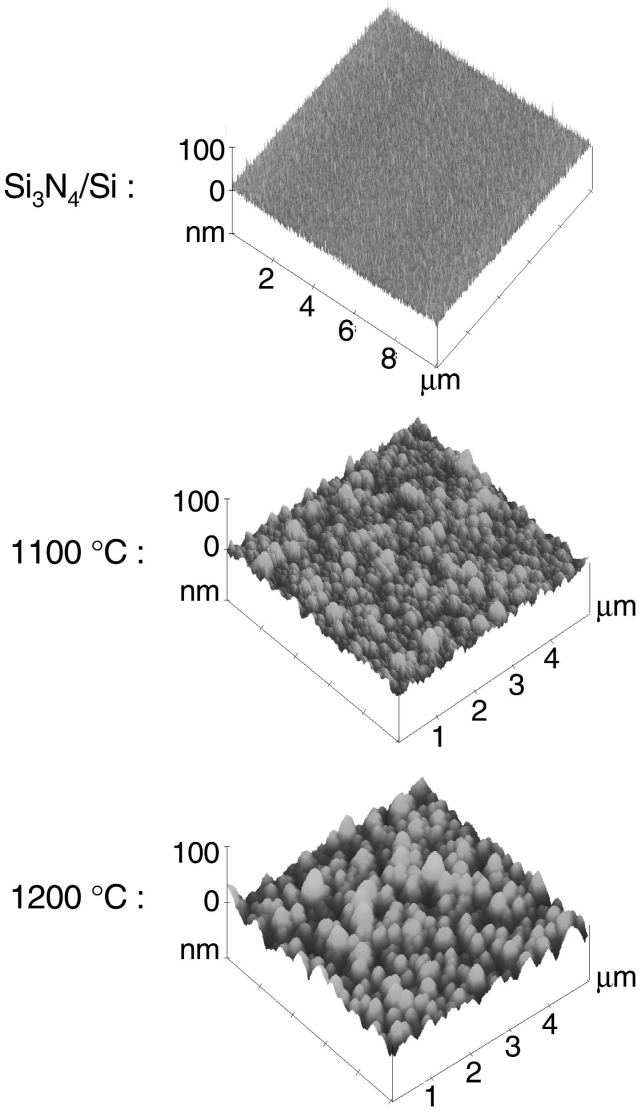


Fig. 10. The AFM micrographs of starting $Si_3N_4$/Si surface and SiC-film surface grown at 1100°C and 1200°C. Flow rates: 30 sccm 3MS and 1 sccm $N_2$.

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